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# Flame Structure Studies of a Lean 20-Torr H<sub>2</sub>/N<sub>2</sub>O/Ar Flame Employing Molecular Beam Mass Spectrometry

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13. ABSTRACT (Maximum 200 words) In this report, we present the results of our continued experimental efforts to investigate the structure of low-pressure, burner-stabilized H <sub>2</sub> /N <sub>2</sub> O/Ar flames. Coated Pt/Pt-Rh(10%) thermocouples and molecular beam sampling followed by quadrupole mass spectrometric detection (MB/MS) were employed to obtain temperature and species mole fraction profiles, respectively, in a fuel lean ( $\Phi=0.64$ ), 20-torr H <sub>2</sub> /N <sub>2</sub> O/Ar flame. Species profiles of H <sub>2</sub> , N <sub>2</sub> O, N <sub>2</sub> , H <sub>2</sub> O, NO, O <sub>2</sub> , H, O, and OH are compared to profiles generated using a detailed flame kinetic model. This provides a stringent test of the detailed chemical flame model and chemical mechanism for the H <sub>2</sub> /N <sub>2</sub> O/Ar system which is presently being developed at the U.S. Army Research Laboratory to enhance the understanding of nitramine propellant combustion and decomposition.					
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## 1. INTRODUCTION

The  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flame/reaction system has been the subject of a number of previous studies which have included shock tube (Henrici and Bauer 1969; Dean 1976; Dean, Steiner, and Wang 1978; Pamidimukkla and Skinner 1985; Hidaka, Takuma, and Suga 1985a, 1985b) and bulb (Baldwin, Gethin, and Walker 1973; Baldwin, et al. 1975) experiments, as well as atmospheric pressure (Cattolica, Smooke, and Dean 1982; Vanderhoff et al. 1977) and low pressure (Balakhnine, Vandooren, and Van Tiggelen 1977; Kohse-Hoinghaus et al. 1988) flame experiments. This system has received considerable attention because it is one of the simplest chemical systems in which nitrogen chemistry can be investigated and, therefore, has important implications in understanding  $\text{NO}_x$  pollutant formation and nitramine propellant combustion and decomposition. Several of the earlier flame studies focused on measuring the concentration of major stable reactants and products. It was clear, however, that a more comprehensive measurement of reactants and products, as well as reactive radical intermediates, was needed in order to construct a detailed chemical mechanism for the  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  system. In a study by Balakhnine, Vandooren, and Tiggelen (1977), all stable species profiles and several radical species profiles were measured in a 40-torr lean (31.4%  $\text{H}_2$ ,  $\Phi = 0.46$ )  $\text{H}_2/\text{N}_2\text{O}$  flame using the molecular beam sampling/mass spectrometric detection (MB/MS) technique. This study was the first comprehensive investigation of this system and pointed out the main features of the combustion mechanism. Experimental results which have been presented for a 20-torr stoichiometric  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flame (Howard, Sausa, and Miziolek 1991) include a thermocouple temperature profile and relative species profiles for  $\text{H}_2$ ,  $\text{N}_2\text{O}$ ,  $\text{N}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{NO}$ ,  $\text{O}_2$ ,  $\text{H}$ ,  $\text{O}$ ,  $\text{OH}$ , and  $\text{NH}$ . The experimental stable species profiles have since been quantified into absolute species concentration profiles and a detailed chemical mechanism has been constructed based on an extensive examination of the literature concerning gas phase nitrogen chemistry (Sausa et al. 1992). The reliability of the detailed flame model was demonstrated by the overall agreement between the experimental and calculated species profiles for the stoichiometric  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flame.

Experimental conditions where the chemistry of the  $\text{H}_2/\text{N}_2\text{O}$  system was significantly changed were sought in order to provide a more rigorous test of the model. These conditions were met in the earlier study by Balakhnine, Vandooren, and Tiggelen (1977) involving a low-pressure lean  $\text{H}_2/\text{N}_2\text{O}$  flame. However, several inconsistencies concerning the production of  $\text{NO}$  and  $\text{O}_2$  (Anderson and Faust 1992) arose when the experimental and calculated results were compared. As a result, we have undertaken a comprehensive experimental investigation of a lean  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flame to resolve these inconsistencies and provide further validation of the detailed chemical model.

Presented below are temperature and species profiles measured in a 20-torr fuel lean ( $\Phi = 0.64$ )  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flame. Stable and radical species mole fraction profiles were measured using molecular beam sampling followed by quadrupole mass spectrometric detection and compared to modeling results. A detailed description of the chemical mechanism will appear in the literature (Sausa et al. 1992).

## 2. EXPERIMENTAL

The experimental apparatus utilized in this study consists of a molecular beam sampling/triple quadrupole mass spectrometer (MB/MS) coupled to a low pressure burner chamber. This system has been previously described in detail in the literature (Sausa et al. 1992; Howard et al. 1992), however, a brief description of the salient points relevant to the present study follows. The  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flame was supported on a McKenna flat-flame burner housed in a cylindrical stainless steel vacuum chamber maintained at 20 torr. Commercial high-purity grade gases were metered with MKS mass flow controllers, which were cross checked with a GCA Precision Scientific wet test meter. Initial volumetric flow rates for  $\text{H}_2$ ,  $\text{N}_2\text{O}$ , and Ar were 1.25, 1.95 and 1.40 standard liters per minute, respectively, resulting in a stoichiometry of 0.64.

The flame gases are sampled through a 200- $\mu\text{m}$ -diameter orifice in a conical quartz sampler and supersonically expanded into the first differential vacuum chamber of the triple quadrupole mass spectrometer system. Collimating the expanding gases with a second 2-mm-diameter skimmer forms a molecular beam. The collisionless environment of the molecular beam insures that the chemistry is essentially frozen (i.e., chemical reactions are quenched and radical recombination is inhibited). As a result, stable reactants and products, as well as highly reactive radical intermediates, can be probed. The molecular beam is modulated by a tuning fork chopper and directed into the ionization region of an Extrel C-50 triple quadrupole mass analyzer. Only a single quadrupole was utilized in the present study. The modulated electron multiplier current intensity at each ion mass-to-charge ratio is phase sensitively detected with a lock-in amplifier to discriminate against background gases and accommodate signal averaging to increase sensitivity.

An ionization energy of  $17.0 \pm 0.3$  eV and an electron current of  $0.20 \pm 0.01$  mA was maintained for all of the species profiles measured in this study, with the exception of the oxygen atom. The ionization energy while measuring the O atom profile was lowered to 15.1 eV, just below the appearance potential for forming  $\text{O}^+$  from  $\text{N}_2\text{O}$  (Collin and Lossing 1958).

The temperature profile for the  $\Phi = 0.64$   $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flame was measured with a coated Pt/Pt-Rh(10%) fine wire thermocouple. Seventy-five micron diameter platinum and platinum with 10% rhodium wires were spot welded together to form a thermocouple junction. Coating the wires with a beryllium oxide (15%)/yttrium oxide mixture according to the procedure described by Kent (1970) eliminated surface catalytic effects which can result in erroneous measured temperatures. The temperature profile presented in Figure 1 was then obtained by correcting the measured temperatures for radiation losses (Sausa et al. 1992; Peterson 1981) from the 201- $\mu\text{m}$ -diameter coated thermocouple junction. The peak temperature (2,161 K) is similar to that measured in the stoichiometric  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flame (2,178 K) (Sausa et al. 1992). The adiabatic flame temperatures for the  $\Phi = 0.64$  (2,422 K) and  $\Phi = 1.00$  (2,494 K)  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flames determined from a NASA-Lewis equilibrium calculation (Svehla and McBride 1973) are also of similar magnitude. The solid line connecting the experimental points is a fit to a sigmoidal function with an extrapolated burner surface temperature of 425 K. The fitted temperature profile was used as input in the model. Another option is available in which the energy equation is solved and the temperature is calculated in addition to the species profiles. This option was not exercised because experimentally there are considerable heat losses other than to the burner surface which, at present, cannot be properly accounted for in the model resulting in unreasonable calculated temperature profiles. As a result, the option of using the temperature profile as an input into the model was chosen (Sausa et al. 1992).

### 3. RESULTS

Absolute mole fractions of the stable species in the  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flame were calibrated by comparing the ion current intensities at each mass-to-charge ratio measured in the flame gases to those measured in a known gas mixture. The relationship between the signal current intensity and the mole fraction can be expressed in terms of a sensitivity factor which is a function of the ionization cross section and several instrument parameters. Peeters and Mahnen (1973) have reported that the temperature dependence of the species sensitivity factor is the same for all species. The ratio of sensitivity factors of any two species, therefore, remains constant at every point in the flame. Using the appropriate sensitivity factors determined under ambient conditions, it is possible to relate relative ion current intensities measured at different points in the flame to relative mole fractions in the flame.

Sensitivity factors for  $\text{H}_2$ ,  $\text{N}_2\text{O}$ ,  $\text{N}_2$ ,  $\text{NO}$ , and  $\text{O}_2$  relative to Ar were determined by calibrating gas mixtures using nearly identical mass spectrometer operating conditions as those used during the flame measurements. The sensitivity factor for  $\text{H}_2\text{O}$  was not determined in this manner because of the difficulty

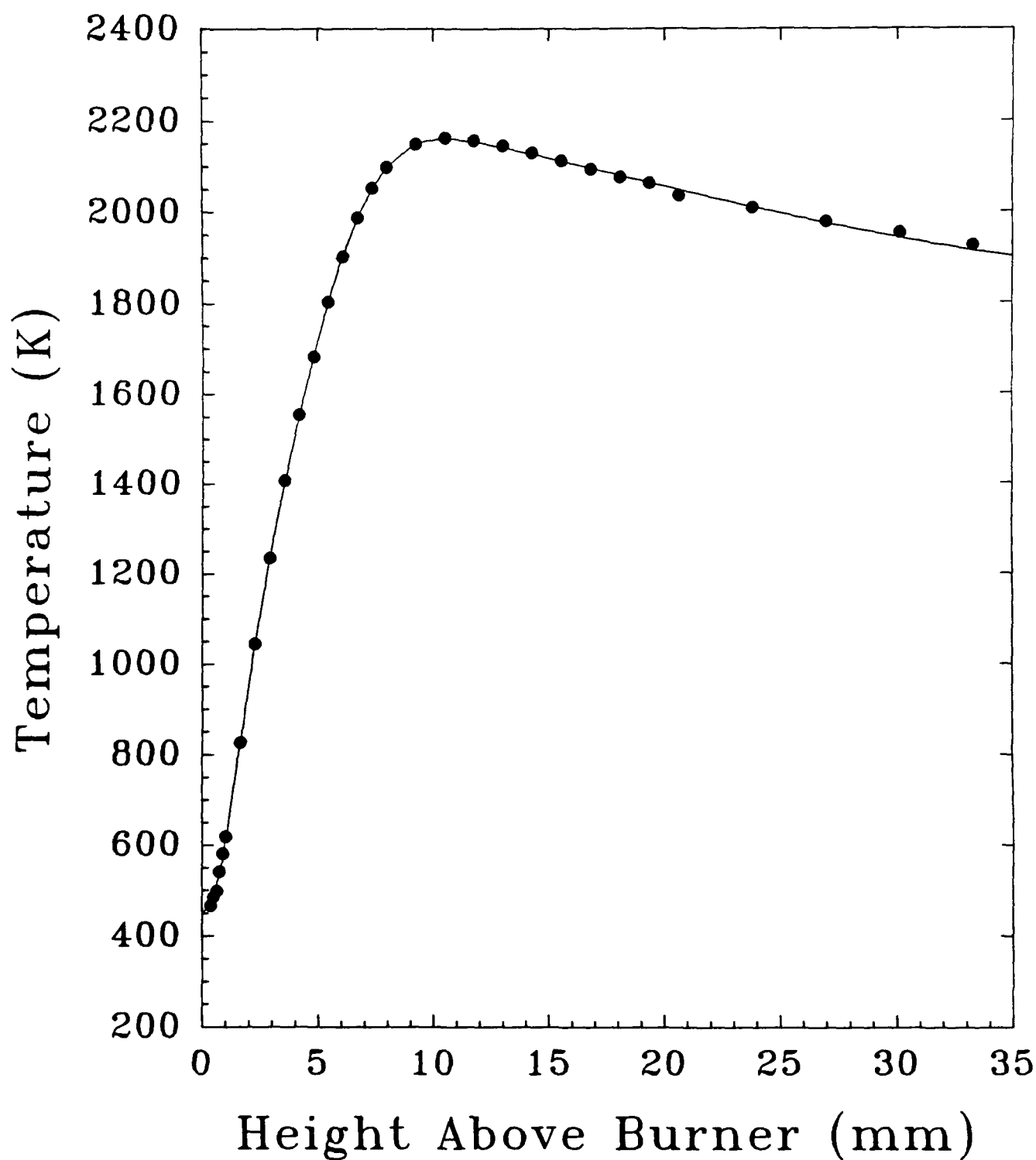


Figure 1. Thermocouple temperature profile of the lean ( $\Phi = 0.64$ )  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  flame. The solid line is a fit to a smooth sigmoidal function.

of reliably introducing a precise amount of water vapor into the system. The  $H_2O$  signals measured in the flame, however, were quantified by equating the ratio of N/O in the premixed gases to the ratio of N/O in the burnt gases (Vandooren, Branch, and Van Tiggelen 1992). The resulting expression for the partial pressure of  $H_2O$  vs. the partial pressure of Ar in the burnt gases, where the  $H_2O$  signal is constant, is:

$$\left(\frac{P_{H_2O}}{P_{Ar}}\right) = \left(\frac{P_{N_2}}{P_{Ar}}\right) - \frac{1}{2}\left(\frac{P_{NO}}{P_{Ar}}\right) - 2\left(\frac{P_{O_2}}{P_{Ar}}\right).$$

This expression was derived assuming that the mole fractions of the radical species are comparatively small.

Since the sum of the mole fractions is unity, the mole fractions of the individual stable species can be determined by dividing the partial pressures of the stable species relative to the partial pressure of Argon by the sum of the partial pressures of the stable species relative to Ar plus one (the partial pressure of Ar relative to itself is one). The mole fraction of Ar was obtained by normalizing the relative Ar ion current intensity to the mole fraction of Ar in the premixed gases. The above outlined procedure resulted in the absolute species profiles presented in Figures 2 and 3. The errors associated with the sensitivity factors results in a relative estimated error of  $\pm 10\%$  for each of the absolute species profiles.

As mentioned above, the mole fractions of the radical species, in particular, H, O, and OH, are assumed small in comparison to the mole fractions of the stable species. Unfortunately, it is not possible to determine the absolute mole fractions of the radical species by direct calibration. It is possible, however, to quantify the relative radical species profiles if the  $H_2$ - $O_2$  reactions are assumed to be in partial equilibrium in the burnt gases of the flame. In the present study, however, the relative H, O, and OH concentrations presented in Figures 4–6 were normalized to the modeling results. A partial equilibrium analysis is planned for future work.

#### 4. DISCUSSION

The impetus for studying a lean  $H_2/N_2O/Ar$  flame was to provide experimental results in order to validate the chemical mechanism for this system being developed in our laboratory (Sausa et al. 1992; Anderson and Faust 1992). Figure 2 displays the major species profiles measured for  $H_2$ ,  $N_2O$ ,  $N_2$ ,  $H_2O$ , and Ar and the accompanying profiles calculated using our detailed flame model (Anderson and Faust 1992). Overall, the computed profiles accurately model the experimental results throughout the entire

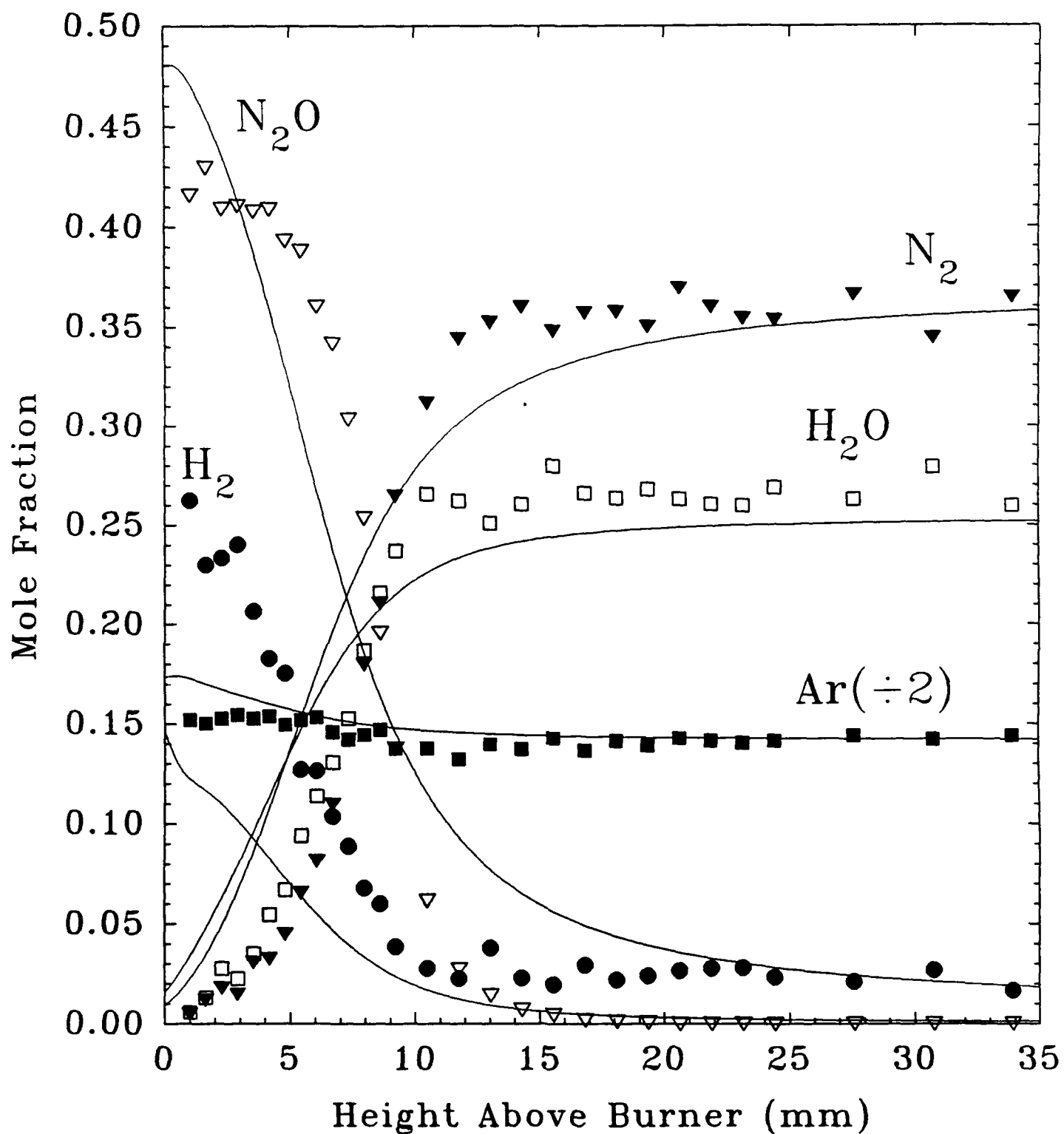


Figure 2. Measured and calculated major species mole fraction profiles for the 20-torr lean ( $\Phi = 0.64$ )  $H_2/N_2O/Ar$  flame.



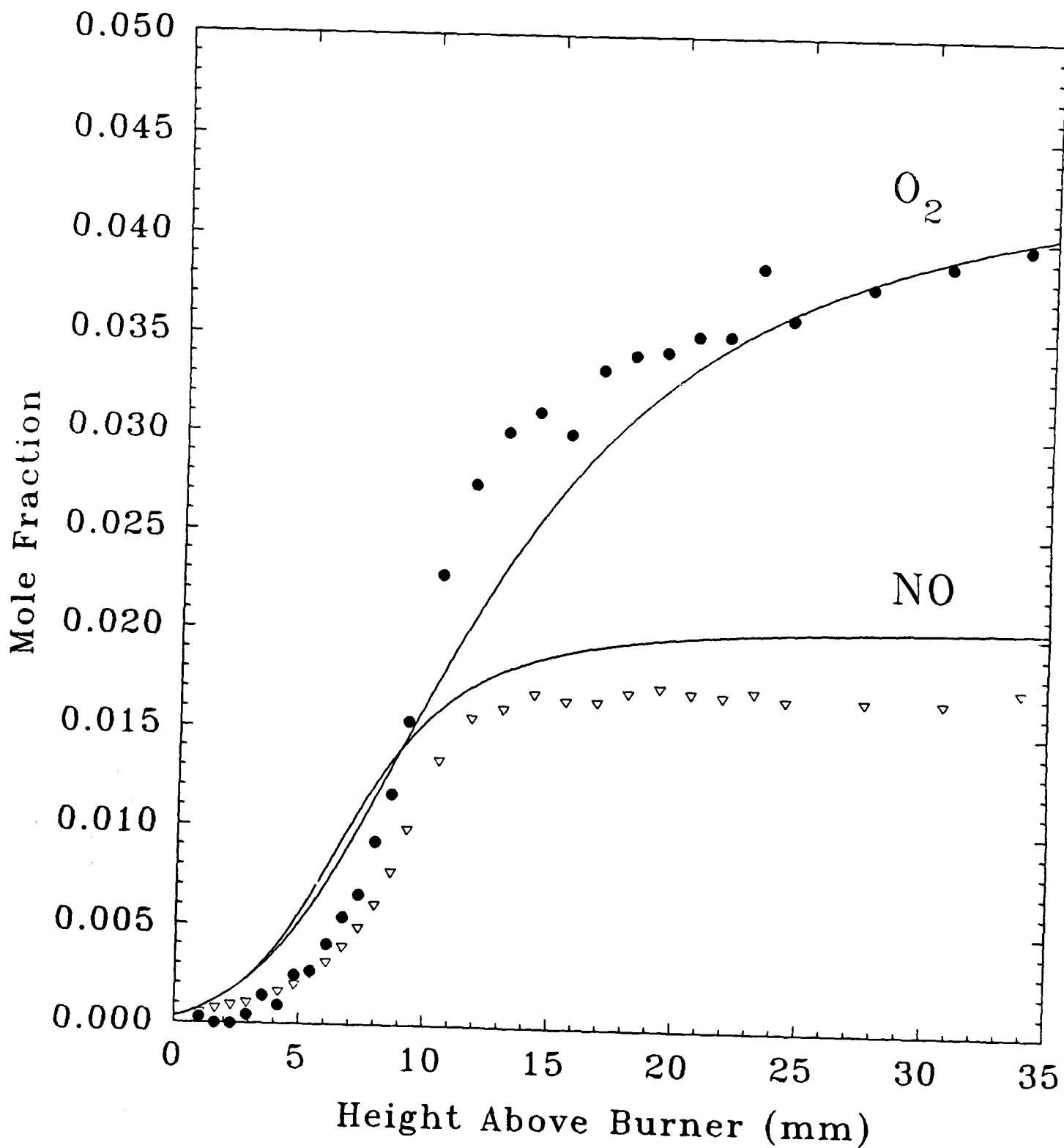


Figure 3. Experimental and calculated NO and  $O_2$  mole fraction profiles in the lean ( $\Phi = 0.64$ )  $H_2/N_2O/Ar$  flame.

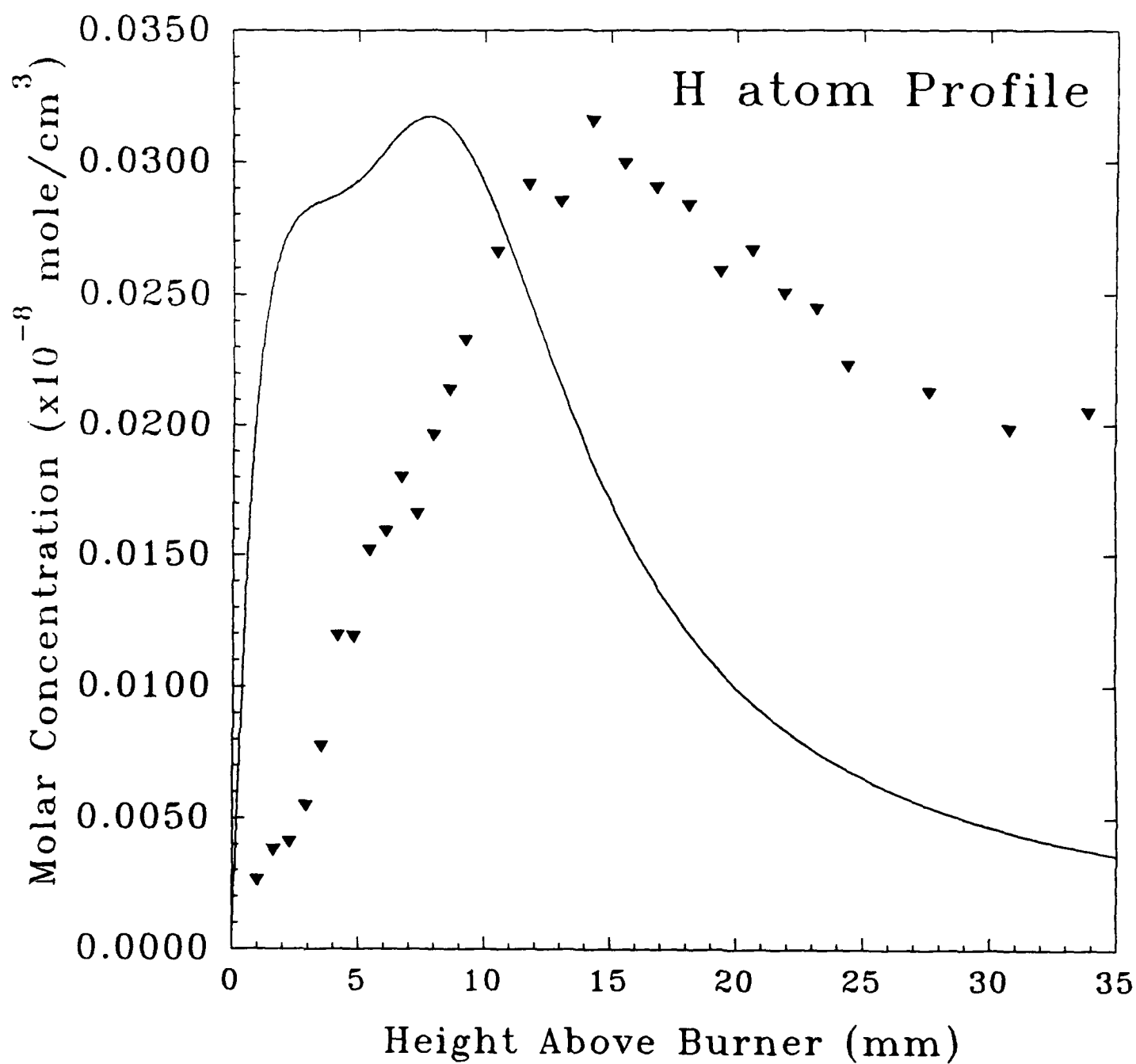


Figure 4. Experimental relative H atom profile normalized to the calculated H atom molar concentration (mole/cm<sup>3</sup>).

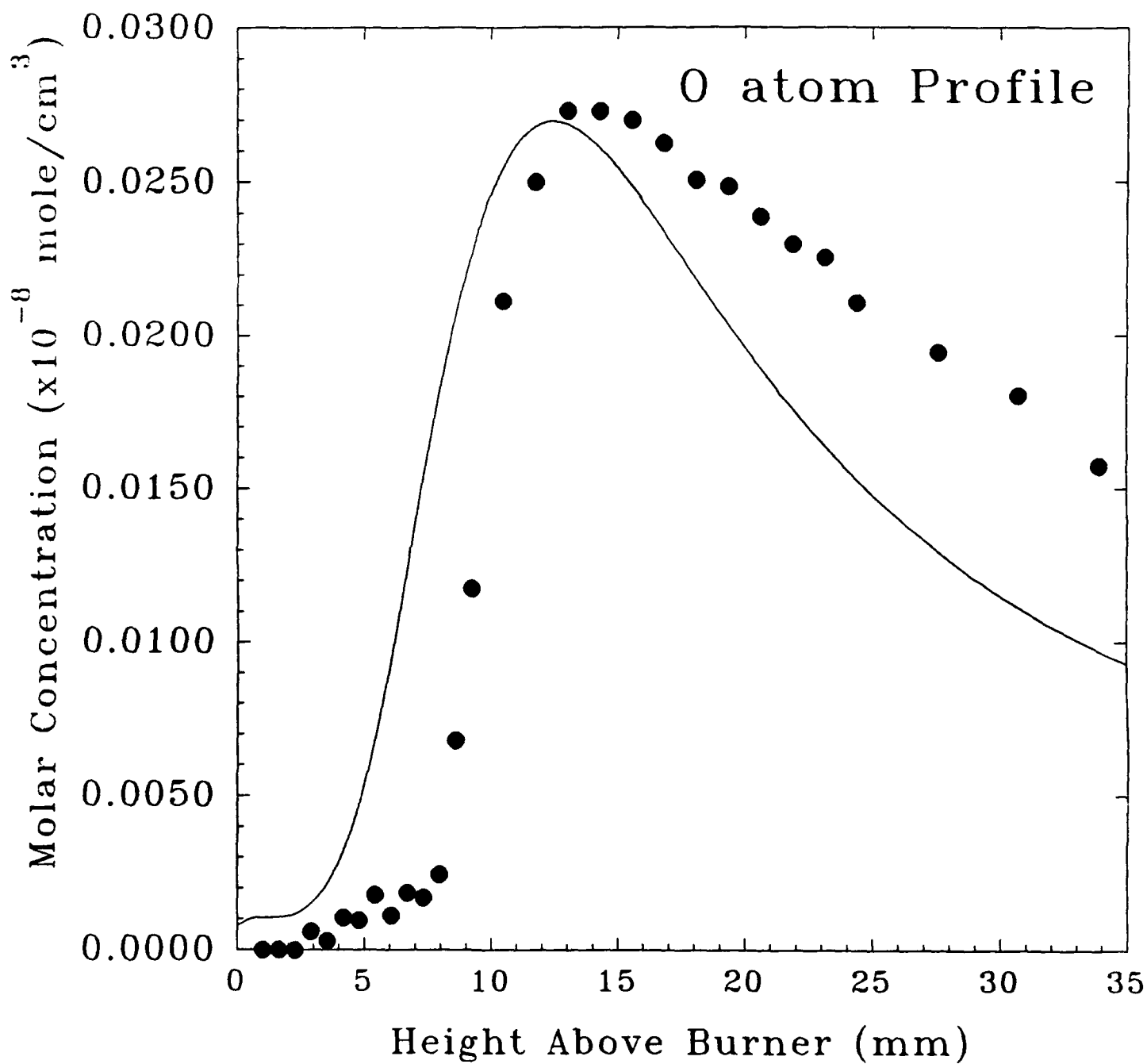


Figure 5. Experimental relative O atom profile normalized to the calculated O atom molar concentration (mole/cm<sup>3</sup>).

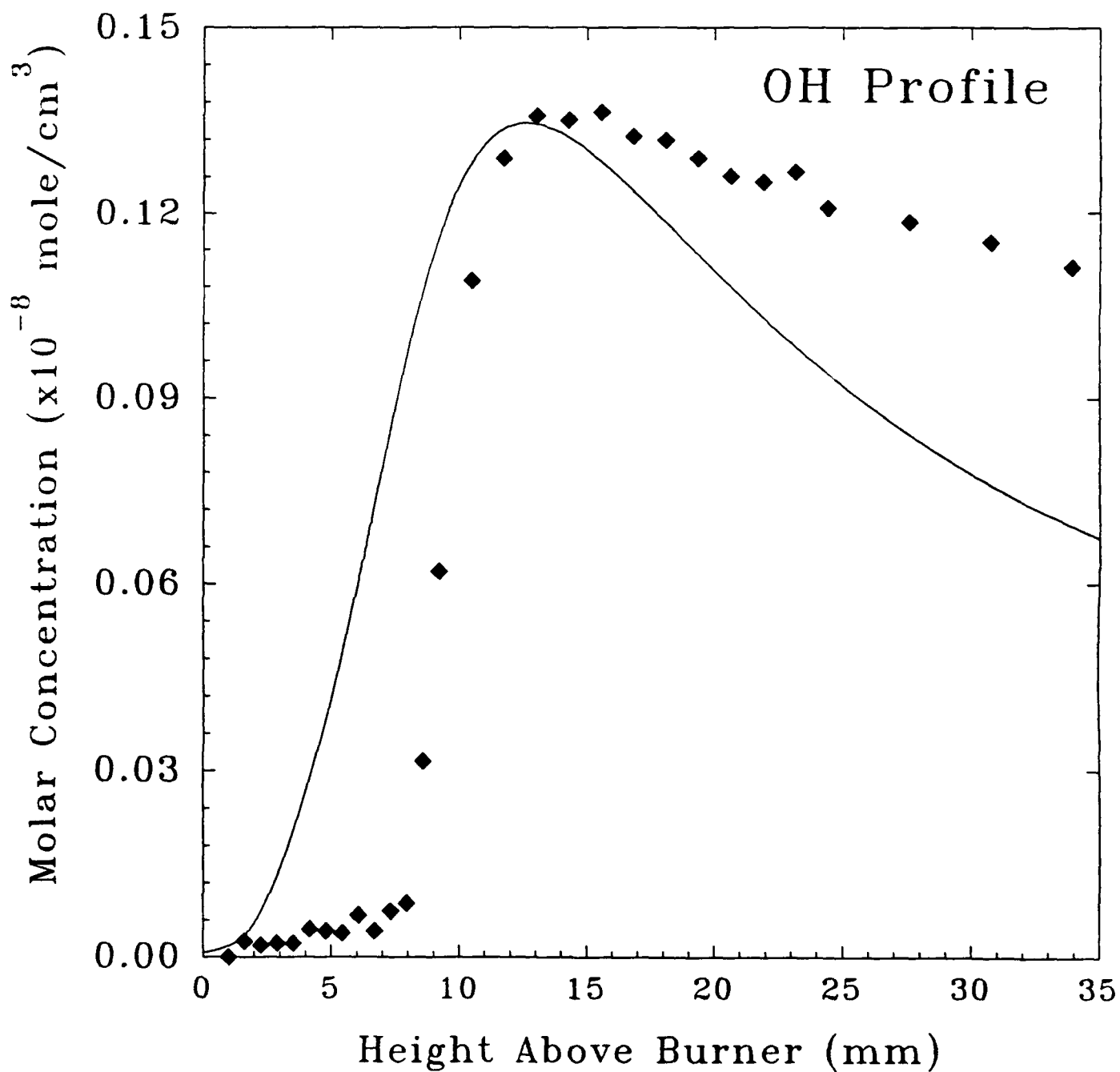


Figure 6. Experimental relative OH profile normalized to the calculated OH molar concentration (mole/cm<sup>3</sup>).

flame. Near the burner surface, in the preheat zone of the flame, the model slightly overpredicts the  $\text{N}_2\text{O}$  mole fraction and underpredicts the  $\text{H}_2$  mole fraction. The discrepancy in the  $\text{N}_2\text{O}$  profiles is within the estimated uncertainty. The difference between the  $\text{H}_2$  profiles is slightly larger and at the present time, this discrepancy is not well understood. The experimental and modeling profiles for the products,  $\text{N}_2$  and  $\text{H}_2\text{O}$ , agree well in the preheat zone. However, the calculated  $\text{N}_2$  and  $\text{H}_2\text{O}$  mole fractions tend to increase more rapidly through the flame front compared to the measured mole fractions. Conversely, the predicted  $\text{N}_2\text{O}$  mole fraction decreases more rapidly through the flame front compared to the experimental results.

Table 1. Experimental and Calculated Stable Species Mole Fractions in the Post Flame Region of the Lean ( $\Phi = 0.64$ )  $\text{H}_2/\text{N}_2\text{O}/\text{Ar}$  Flame Approximately 25 mm Above the Burner Surface

Species	Experiment	Premix Model	NASA/Lewis (Equilibrium)
$\text{H}_2$	0.021	$2.15 \times 10^{-3}$	$1.68 \times 10^{-3}$
$\text{N}_2\text{O}$	0.00	0.0275	$1.99 \times 10^{-8}$
$\text{N}_2$	0.36	0.351	$3.91 \times 10^{-1}$
$\text{H}_2\text{O}$	0.26	0.250	$2.47 \times 10^{-1}$
$\text{O}_2$	0.036	0.0365	$6.78 \times 10^{-2}$
NO	0.034	0.0402	$3.07 \times 10^{-3}$

A comparison of the experimental and predicted mole fractions of the major stable species determined in the post flame region, 25 mm above the burner surface, is presented in Table 1. Also included for comparison are the equilibrium mole fractions of these species determined from a NASA-Lewis chemical equilibrium calculation (Svehla and McBride 1973) using a temperature of 2,000 K which corresponds to a height of 25 mm above the burner. The model predicts a significant  $\text{N}_2\text{O}$  mole fraction whereas, experimentally, no appreciable  $\text{N}_2\text{O}$  signal is observed. The equilibrium calculations also suggest that the  $\text{N}_2\text{O}$  mole fraction should be vanishingly small. A measurable amount of  $\text{H}_2$  in the post flame region is observed experimentally, however, the model and the equilibrium calculations predict that  $\text{H}_2$  is almost completely consumed in the post flame gases. The experimental and modeling results for  $\text{N}_2$  and  $\text{H}_2\text{O}$  in the post flame region are in excellent agreement.

Presented in Figure 3 are the calculated and experimental mole fraction profiles for NO and O<sub>2</sub>. Again, the experiment and the model agree well throughout the entire flame. The overall shape of the experimental and calculated NO mole fraction profiles agree well while the experimental O<sub>2</sub> mole fraction appears to increase more sharply through the flame zone than the calculated O<sub>2</sub> mole fraction. The final O<sub>2</sub> mole fraction is accurately predicted by the model, however, the equilibrium O<sub>2</sub> mole fraction is slightly higher than predicted by the model and measured experimentally. The final predicted NO mole fraction is slightly larger than the experimental NO mole fraction. According to the results of the equilibrium calculations, however, the NO mole fraction is considerably smaller than the model prediction suggesting that N<sub>2</sub>O combustion does not completely proceed to the final products N<sub>2</sub> and H<sub>2</sub>O. Formation of NO in the H<sub>2</sub>/N<sub>2</sub>O system has been discussed in the literature (Sausa et al. 1992) and has been shown to be very important in understanding N<sub>2</sub>O oxidation and pollutant formation.

The experimental and calculated profiles for the H, O, and OH radicals are presented in Figures 4–6, respectively. As discussed above, the relative profiles were normalized to the modeling results because an independent measurement of the absolute concentrations of these unstable species was not attempted in this study. The general shapes of the calculated O and OH profiles agree well with the experimental results. The predicted H atom concentration is considerably higher near the burner surface than observed experimentally. Each of the experimental radical species profiles peaks at distances further above the burner surface than the model predicts. This discrepancy is greatest in the H atom profiles. The experimental O and OH profiles are both relatively flat near the burner surface and tend to increase rapidly through the flame zone, which begins approximately 8 mm above the burner surface. The calculated O and OH profiles tend to rise more steeply closer to the burner surface than the experimental results. Given the overall agreement of the experimental and calculated results for the stable species, it is difficult at this time to ascertain whether these discrepancies result from perturbations to the measured profiles induced by the quartz sampling probe or subtle inefficiencies in the model. Previous studies (Sausa et al. 1992; Howard et al. 1992) have indicated, however, that the extent of the perturbations to the flame caused by the quartz sampling probe are small.

The NH radical was not observed under the present experimental conditions even though the model predicts that the NH concentration should be larger than in the previous study of the stoichiometric H<sub>2</sub>/N<sub>2</sub>O/Ar flame (Sausa et al. 1992) where NH was detected. The NH concentration in the present study is presumably below the detection limit of the apparatus and may indicate subtle problems with the model.

Both the model and the experiment will be examined in the future to minimize the apparent inconsistencies between the experiment and the model.

## 5. CONCLUSIONS

Presented in this paper are the experimental mole fraction profiles for  $H_2$ ,  $N_2O$ ,  $N_2$ ,  $H_2O$ ,  $NO$ ,  $O_2$  and the species concentration profiles for  $H$ ,  $O$ , and  $OH$  measured in a fuel lean ( $\Phi = 0.64$ )  $H_2/N_2O/Ar$  flame stabilized at 20 torr. The mole fractions of the stable species were determined by direct calibration of the mass spectrometer signals. The relative species profiles of the unstable radical species  $H$ ,  $O$ , and  $OH$  were normalized to the modeling results. These results provide a stringent test of the model for the  $H_2/N_2O/Ar$  chemical system being developed in our laboratory (Sausa et al. 1992; Anderson and Faust 1992). Overall, acceptable agreement has been achieved between the experimental and calculated mole fractions.

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